

THE PREPARATION OF THE ^{119}Sb MOSSBAUER SOURCE

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Summary

This paper introduces the preparation of the ^{119}Sb Mossbauer Source by irradiating natural Sn metal with 32Mev α -particles from cyclotron using (α, xn) reaction, $x \leq 4$.

After ^{119}Sb was chemically separated from Te, electroplating, diffusion and chemical procedure were used for making $^{119}\text{Sb}(\text{Sb})$ and $^{119}\text{Sb}_2\text{O}_3$ Mossbauer source, respectively. The measurement was performed on a self-made Mossbauer spectrometer. Moreover, the parabola-Lorentzian least square fits were used to analyze Mossbauer spectra both in single line and double line in consideration of a quadrupole split of 0.50(2)mm/s for absorber SnO_2 . The isomer shifts are -1.79(6)mm/s and 0.097(70)mm/s for $^{119}\text{Sb}(\text{Sb})$ and $^{119}\text{Sb}_2\text{O}_3$, respectively.

The results show that the chemical process we used is suitable for separating Sb, the diffusion temperature 535°C ($T_d = 0.85T_m$, T_m is melting temperature) and temperature-keeping time are adequate, too.

Introduction

The Ion-implantation Mossbauer spectroscopy has been developed rapidly in recent decade, it is an important method for investigating microscopic variation of the lattice structure due to ion doping. With this method, the lattice damage caused by ion irradiation, the sites of doping atoms in the lattice and their aggregation can be observed and the dynamics of the defect and impurity atoms can be studied in different temperature as well.

It has been proved that the radioactive ion-implantation is the most effective method in ion-implantation Mossbauer spectroscopy.

The impurity ions implanted play both the projectiles which cause lattice damage and the probe. The requirement for the dose of radioactive ions, therefore, can be considerably reduced, with ^{119m}Sn , ^{119}Sb , and ^{57}Co , for instance, several hundred nano curie activities are only needed. Under the condition of low dose implantation the overlap of cascade collision can be neglected, therefore, the study of individual cascade turns out to be possible.

Now the problem is how to search for a suitable radioactive Mossbauer nuclide as an implanted source. With large recoil-free fraction, high extraction efficiency for isotope separator as well as short life-time for preventing separator from contamination are necessary to the source. The ^{119}Sb , ^{119m}Sn , ^{133}Xe , ^{129}Te , ^{57}Co , and ^{83}Kr sources (1-4) are widely used, especially ^{119}Sb is adopted by university of Aarhus in Denmark and University of Groningen in the Netherlands, it is produced by reaction of (α, n) , $(\alpha, 2n)^{(3)}$ and $(p, 3n)^{(5)}$. The ^{119}Sb with the half-life of 38h decays to ^{119m}Sn by electron capture. In room temperature it has a reasonable recoil-free fraction both in metal and semiconductor. This paper introduces the preparation of the Mossbauer source of $\text{Sb}(\text{Sb})$ and Sb_2O_3 .

^{119}Te isomer (4.7d) was produced by irradiating natural Sn metal with 32Mev α -particles from cyclotron of Shanghai Institute of Nuclear Research. After ^{119}Sb was chemically separated from Te, the electroplating, diffusion and chemical processing were used to make these Mossbauer sources, respectively, and the quality of them was examined by γ -ray spectra and Mossbauer spectra.

Preparation

A piece of natural Sn(99.999%) was rolled to a flat disk and molten on the surface of a silver plate at about 250°C. The thickness of the Sn target was about 200mg/cm² which was kept by cutting the extra part using lathe. The target was irradiated with 32Mev α -particles with water cooling. The beam current was 5 μ A and the total dose was 100MAh. Except for ¹¹⁷Te and long life-time ¹²¹Te the main product was ¹¹⁹Te. Figure 1(a) shows typical γ -ray spectra detected by Si(Li) detector before chemical separation.

The ¹¹⁹Te produced by irradiating natural Sn metal contained both ^{119g}Te (T_{1/2}=16h) and ^{119m}Te(4.7d). They decay to ¹¹⁹Sb through ^{119g}Te $\xrightarrow{\beta^+ \text{ EC}}$ ¹¹⁹Sb and ^{119m}Te $\xrightarrow{\text{EC}}$ ¹¹⁹Sb, respectively. Therefore Te has to be first separated from the target, then Sb was separated from Te.

The irradiated Sn target was separated from the Ag plate by dissolving it in concentrated hot HCl. A black residue which carries ¹¹⁹Te was deposited. The Te was extracted from the residue with concentrated HNO₃ (8 drops)+1ml HCl in which 5mg Te carrier(99.999%) was added. Some concentrated HCl was added to remove HNO₃ by fuming. The elemental Te was precipitated from 4N HCl with SO₂ gas, then evaporating the liquid in a water bath at $\leq 100^\circ\text{C}$ for 40 minutes. The Te precipitation was repeated several times in order to reduce the contamination with Sn. After 4 days, the 38-h ¹¹⁹Sb activity was extracted from the purified Te using the same process as mentioned above except that 3mg Sb carrier was added in stead of Te carrier. When Te reduced with SO₂ gas was extracted the liquid was evaporated by infrared heating to a suitable volume for use. Figure 1(b) shows the γ -ray spectrum underwent above chemical separation. The remaining Te can be separated for ¹¹⁹Sn after another 4 days growth.

To prepare the Mössbauer source Sb(Sb), the purified Sb powder (99.999%) was molten on a copper bath in depth of 0.2mm in H₂ atmosphere, then mounted in the electroplated bath. The electrolyte was 8% H₂SO₄, the area electroplated was 5mm in diameter. Electro-

plating lasted ten hours at current of 20mA and at temperature of 50°C. The electroplated sample was annealed in H₂ atmosphere at temperature of 535°C in 30 minutes, then cooled down slowly.

For preparing the Mössbauer source Sb₂O₃, several drops of SbCl₃ solution were taken from the second time separated solution. After drying several ml H₂O was added, then heating and separating by centrifuge, the white Sb₂O₃ was precipitated from H₂O and NH₄OH solution. Finally the Sb₂O₃ was removed onto a sheet of filter paper through rinsing it several times and drying it by infrared lamp, we got the Mössbauer source of ¹¹⁹Sb₂O₃.

Experimented Results and Discussion

The Mössbauer spectra were measured by a self-made Mössbauer spectrometer with constant acceleration. The NaI(Tl) scintillator was used to detect the γ -rays, a sheet of Pd film with thickness of 0.04mm was inserted between source and absorber to reduce the 25.3Kev characteristic X-rays of Sn atoms. Figure 2 shows the Mössbauer spectra of ¹¹⁹Sb(Sb) and ¹¹⁹Sb₂O₃. The isomer shifts, linewidths and relative transmission showed in Table 1. They were obtained by least square fitting of Lorentzian lines. The absorber SnO₂ with a small quadrupole splitting of 0.5(2)mm/s(6) was used. So the spectra were not only fitted with single line but also double line taking into account this splitting.

Table 1 also shows the results obtained by using Ca ¹¹⁹SnO₃ source which was imported from Radiochemical centre England Amersham. The result pointed out that the linewidths of the ¹¹⁹Sb(Sb) and ¹¹⁹Sb₂O₃ are narrower than that of CaSnO₃ and the transmission rate for ¹¹⁹Sb(Sb) is larger than that of CaSnO₃, although the Debye temperature (210°C) of Sb(Sb)(7) is less than the values of Ca¹¹⁹SnO₃. It was also confirmed that the chemical process we used is suitable for separating Sb from γ -ray spectra of figure 1. After chemical procedure the intensity of the characteristic X-ray of Sb at energy of 26.4Kev was greatly reduced, apparently there was no Te characteristic X-ray appeared in source of

$\text{Ca}^{119\text{m}}\text{SnO}_3$.

From the higher transmission rate for $^{119}\text{Sb}(\text{Sb})$, it proves that the diffusion temperature of 535°C ($T_d=0.85T_m$, T_m is melting temperature) and temperature-keeping time are adequate, too.

Table 1. Hyperfine parameters from least-squares fits to Mössbauer spectra of $^{119}\text{Sb}(\text{Sb})$, $^{119}\text{Sb}_2\text{O}_3$ and $\text{Ca}^{119\text{m}}\text{SnO}_3$.

Mössbauer sources	$^{119}\text{Sb}(\text{Sb})$	$^{119}\text{Sb}_2\text{O}_3$	$\text{Ca}^{119\text{m}}\text{SnO}_3$
Linewidth(s)	0.87(3)	0.89(8)	0.99(5)
(mm/s) (d)	0.77(4)	0.82(3)	0.90(4)
I.S.(mm/s)	-1.79(6)	0.097(70)	0
Transmission rate (%)	9.3	3.3	6.2

Reference

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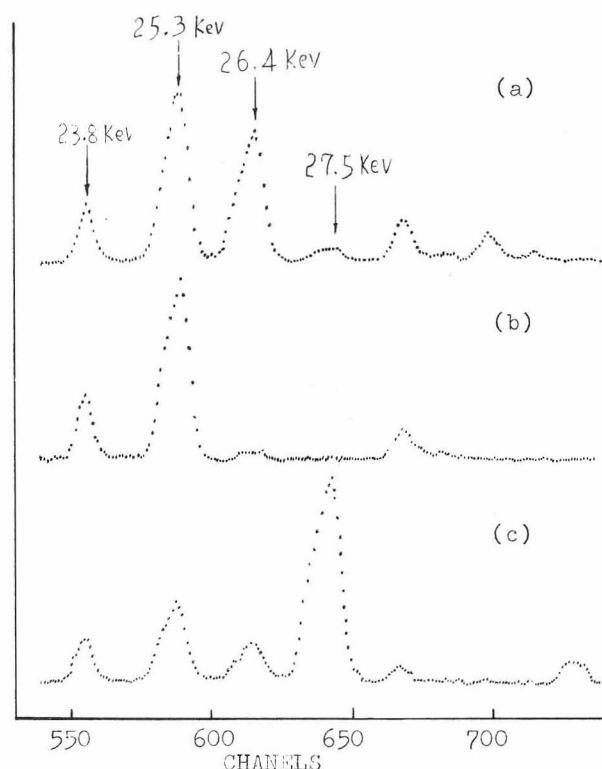


Fig 1. pulse height spectra: (a);(b)- before and after chemically separated, respectively, and (c)- $\text{Ca}^{119\text{m}}\text{SnO}_3$.

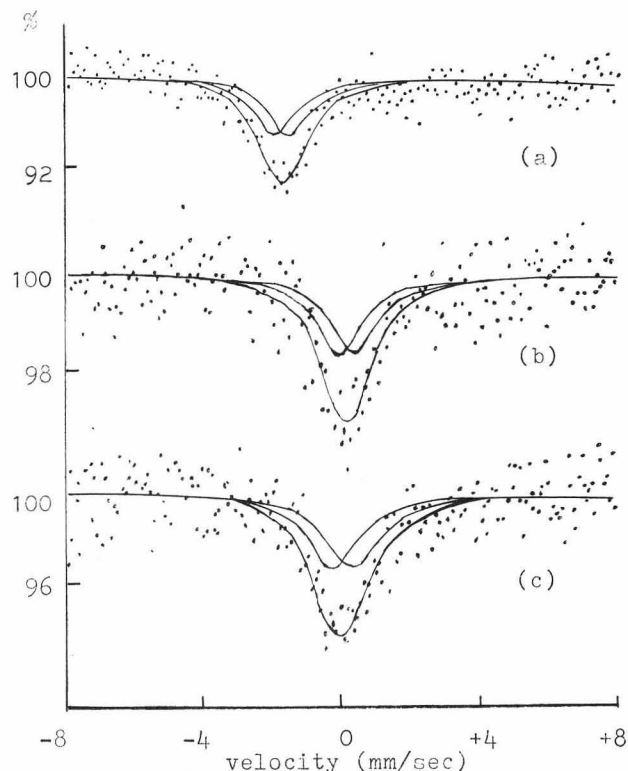


Fig 2. Mössbauer spectra of $^{119}\text{Sb}(\text{Sb})$ (a), Sb_2O_3 (b) and $\text{Ca}^{119\text{m}}\text{SnO}_3$.